ASD-TDR-63-596
Volume II

N

LIGHTWEIGHT THERMAL PROTECTION SYSTEM DEVELOPMENT

Volume II - Materials - Existing Data and Recommended Data Acquisition

Technical Documentary Report No. ASD-TDR-63-596, Volume II

June 1963

AF Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 651G

(Prepared Under Contract No. AF 33(657)-9444
By General Dynamics/Astronautics
San Diego, California;

R. A. Lange, H. Jones, J. M. Ryan, H. Rosenthal, J. Stitt, J. F. Balata, Authors)

ASD-TDR-63-596
Volume II

NOTICES

FOREWORD

This program was initiated by the Materials Applications Division of the Air Force Materials Laboratory, Research and Technology Division, Wright-Patterson AFB, Ohio, with General Dynamics/Astronautics, San Diego, California acting as the Contractor. The work was performed in support of the 651G Program. "Advanced Structures for the Aerospaceplane" with 1/Lt. M. L. Minges of the Materials Engineering Branch, Materials Applications Division serving as RTD project engineer.

General Dynamics/Astronautics Spaceplane Project wishes to express its appreciation to the material suppliers and airframe companies who cooperated in the survey described in this report and also to the Aeronautical Systems Division for prompt action in supplying progress reports from related contracts.

This page intentionally left blank

ABSTRACT

This is the Summary Technical Report covering the first two phases of Contract AF 33(657)-9444 (Lightweight Thermal Protection System Development). It encompasses the task from 1 July 1962 to 1 May 1963. Briefly it contains:

a. A synopsis of a survey of current ideas for lightweight insulative composites, insulation materials, and test equipment available for evaluating thermal protection systems.

b. Preliminary designs of three insulative composites selected for experimental evaluation.

c. The outline of a test program and definition of test equipment to be employed to evaluate the insulative composites selected.

d. Properties of insulation materials.

This technical documentary report has been reviewed and is approved.

W. P. Conrardy

W. P. CONRARDY, Chief,
Materials Engineering Br.
Materials Applications Div.
AF Materials Laboratory

This page intentionally left blank

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

SECTION 1

INTRODUCTION

The long range object of Contract AF 33(657)-9444 is to establish the feasibility of various thermal protection composites applicable to an Aerospaceplane. This is to be accomplished by:

a. Surveying present thoughts and capabilities in the areas of insulative composite developments, insulation materials, and test devices for evaluation of composites.

b. On the basis of information accumulated, selecting several insulative composite designs for experimental evaluation and outlining a test program for such an evaluation.

c. Conducting the test evaluation, correlating test results with analytical performance predictions, and suggesting means of improvement in composite designs.

This TDR covers the Items a and b. The report is divided into two volumes, Volume II (unclassified) containing only material properties, and Volume I (classified SECRET) containing design criteria, description of composites, and outline of test program.

This page intentionally left blank.

SECTION 2

INSULATION MATERIALS

Table 1 and Figures 1 through 5, show insulation materials and properties. The factor of $K \times \rho$ was used for initial comparison of insulation materials. Generally speaking, the heat capacity is near the same for the majority of insulations which are strong candidates for Aerospaceplane composites and since upflight and downflight times are prolonged, the factors of conductivity and density are good criteria for initial material selections.

From the accumulation of data shown in this section, four material candidates were selected for incorporation into the insulative composites proposed in Volume I of this report. These materials are listed below with a brief explanation of the reasons for their choice:

a. Min-K 2000 @ 14 lb/ft^3. This material was chosen for its inherent strength and its low conductivity in the presence of helium gas relative to lower density fibrous insulations. The low conductivity in helium is important for composites which are designed to protect fuel only, or where fuel protection problems tend to predominate over structural temperature problems when fuel is emptied. This is because the helium gas is used to prevent air and water condensation at times when cryogenic fuel exists in the vehicle tanks, and helium gas has about 8 times the conductivity of air. The inherent strength and compression resistance are important for two reasons: First, the insulation does not require encapsulation as is true in the case of loose powder type insulations. Second, the material can take substantial compression loads when used in an internal insulative composite to resist fuel tank pressures. The 14 lb/ft^3 density was selected for minimum $K \times \rho$ product.

b. Micro-Quartz Type II @ 6.2 lb/ft^3. This material was chosen again for inherent strength, and further was selected as the best $K \times \rho$ candidate for use up to 2750°F. The density of 6.2 lb/ft^3 was chosen because of recent data acquired on varying pressure performance in similar fibrous insulations at this density. Ultimately the minimum density of 4.5 lb/ft^3 may prove more desirable. This lower density material is somewhat marginal from the handling standpoint.

c. Hi Temperature Super Insulation (S.I.). This material is far superior to any other in a high vacuum. The prime problem with the material is encasement (as described in Volume I) and the discrepancy between remarkable insulation efficiency and heat losses through mechanical elements in the composites.

d. Micro-Quartz Type II and Platinum Foils @ 6.9 lb/ft^3. By the addition of a very limited number of oxidation resistant radiation barriers, there is a tremendous improvement in the performance of Type II Micro-Quartz. The prime problem here is cost and availability of platinum, or perhaps even more desirable, rhodium foils. Methods of employing substrate materials as carriers for plated or vapor deposited coatings of platinum or rhodium will be investigated in future work.

Micro-Quartz Type II emerged as the most promising insulation material candidate from the standpoint of durability, ease of fabrication, and high temperature capability. There was however, a lack of conductivity data on this material. As a result, contract funds were diverted to Dynatech Corporation, to produce additional conductivity data. Dynatech Corporation was approved by the Air Force for insulation property determination work in the proposed experimental phase of this contract. The proposed data accumulation on Type II Micro-Quartz was therefore a logical early beginning of this work. The data produced by Dynatech was only recently received and is included in Appendix I of this volume. This initial conductivity data is for the 4.5 lb/ft^3 density material.

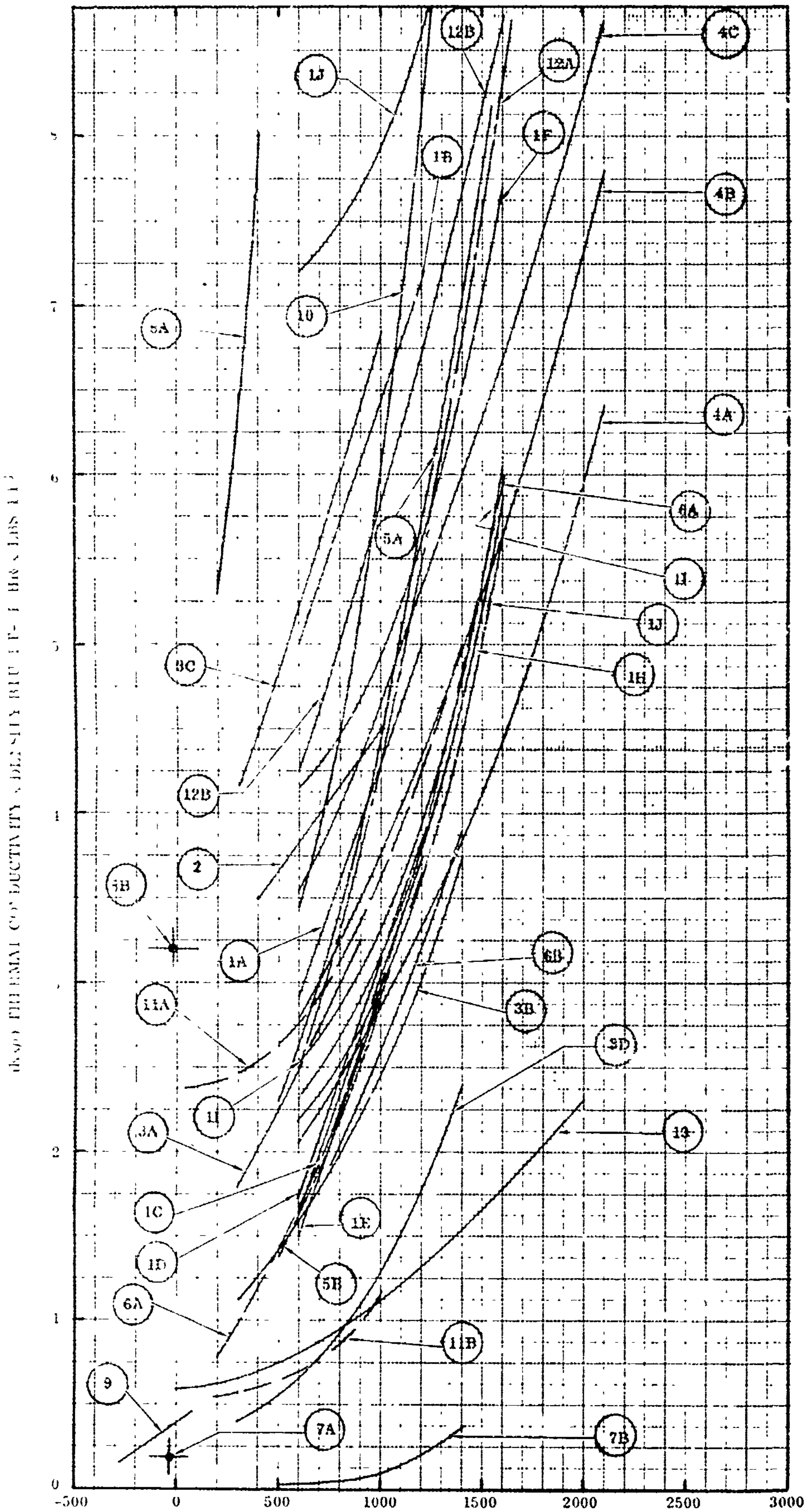

Figure 2   Thermal Conductivity Times Density Versus Temperature

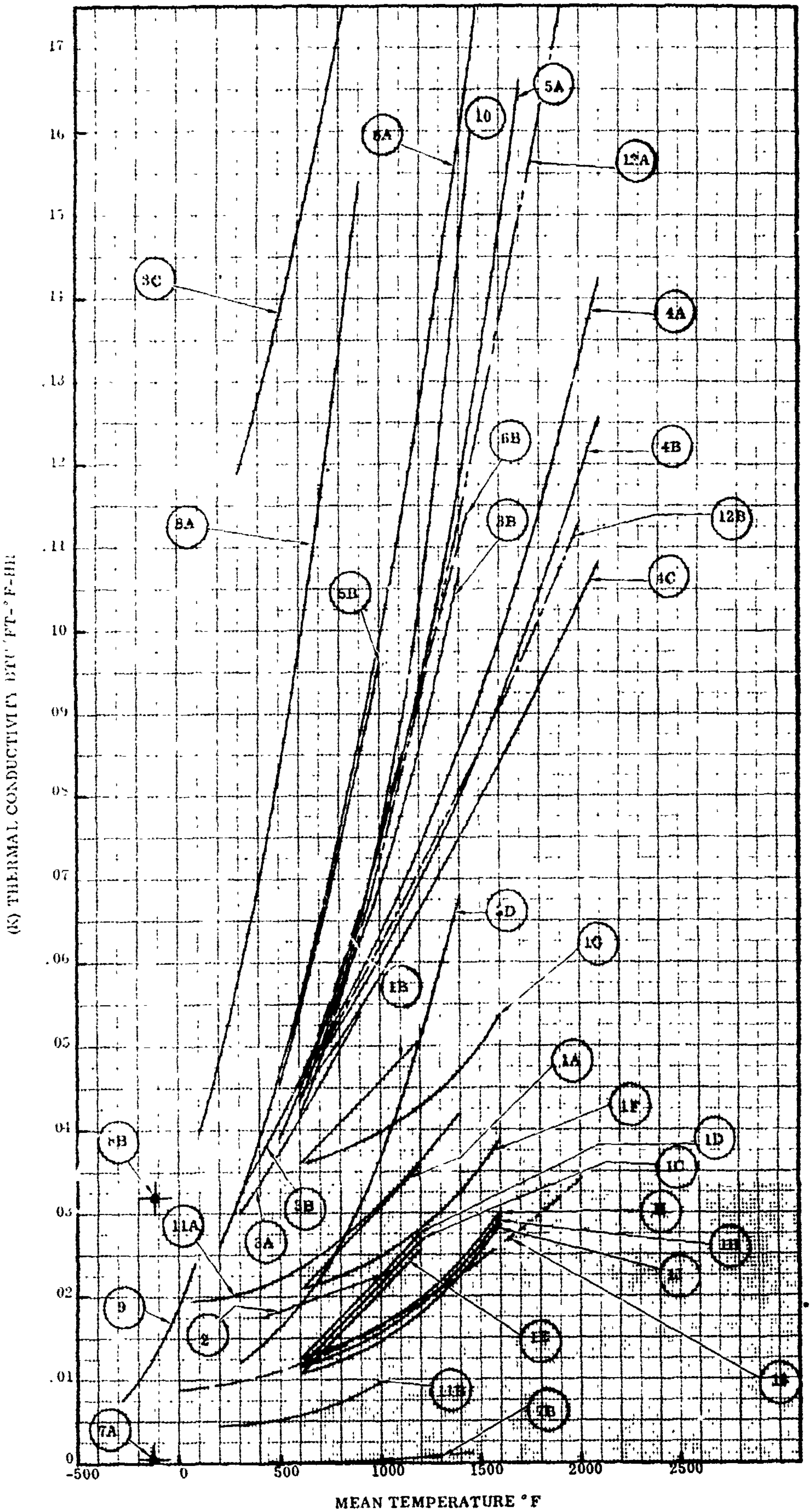

Figure 1. Thermal Conductivity Versus Temperature

Table 1. Insulation Material List

NO.	PRODUCT NAME	SUPPLIER	DENSITY LBS/FT^3	MAX. CONT. SERVICE TEMPERATURE	INTERSTITIAL GAS	GAS PRESSURE (MM HG.)	(K) DATA SOURCE	(C_p) DATA SOURCE
1A	Improved Min-K 2000	Johns-Manville	14	2000°F	Air	760	J/M Survey Data	
1B	Improved Min-K 2000	Johns-Manville	14	2000°F	He	760	J/M Survey Data	
1C	Improved Min-K 2000	Johns-Manville	14	2000°F	Air	55	J/M Survey Data	
1D	Improved Min-K 2000	Johns-Manville	14	2000°F	He	55	J/M Survey Data	
1E	Improved Min-K 2000	Johns-Manville	14	2000°F	Air or He	8	J/M Survey Data	
1F	Improved Min-K 2000	Johns-Manville	20	2000°F	Air	760	J/M Survey Data	J/M Survey Data
1G	Improved Min-K 2000	Johns-Manville	20	2000°F	He	760	J/M Survey Data	
1H	Improved Min-K 2000	Johns-Manville	20	2000°F	Air	55	J/M Survey Data	
1I	Improved Min-K 2000	Johns-Manville	20	2000°F	He	55	J/M Survey Data	
1J	Improved Min-K 2000	Johns-Manville	20	2000°F	Air or He	8	J/M Survey Data	
2	Min-K 1301	Johns-Manville	20	1450°F	Air	760	J/M Survey Data	J/M Survey Data
3A	Micro-Quartz Type I	Johns-Manville	6	2000°F	Air	760	J/M Literature	J/M Survey Data
3B	Micro-Quartz Type I	Johns-Manville	3.5	2000°F	Air	760	J/M Literature	
3C	Micro-Quartz Type I	Johns-Manville	3.5	2000°F	He	760	J/M Lit. & G/D Corr.	
3D	Micro-Quartz Type I	Johns-Manville	3.5	2000°F	Air	High Vacuum	J/M Lit. & G/D Corr.	
4A	Micro-Quartz Type II	Johns-Manville	4.5	2750°F	Air	760	J/M Survey Data	
4B	Micro-Quartz Type II	Johns-Manville	6.2	2750°F	Air	760	J/M Survey Data	
4C	Micro-Quartz Type II	Johns-Manville	8.0	2750°F	Air	760	J/M Survey Data	
5A	Thermoflex RF 600	Johns-Manville	6	2000°F	Air	760	J/M Literature	J/M Survey Data
5B	Thermoflex RF 300	Johns-Manville	3	2000°F	Air	760	J/M Literature	
6A	Refrasil Batt B-100	H. I. Thompson	3	2000°F	Air	760	G/D Fort Worth Rept.	G/D Fort Worth Rept.
6B	Refrasil Batt A-100	H. I. Thompson	3.6	2000°F	Air	760	Hitco Lit.	
7A	S.I.62 Compressed (1 Atm.)	Linde	28		Air	High Vacuum	Linde Lit.	
7B	Hi. Temp. S.I.	Linde	≈18	≈1800°F	Air	High Vacuum	G/D Interpretation of Linde Data	
8A	Foamsil	Pittsburg-Corning	11	2200°F	Air	760	Pitt-Corning Lit.	
8B	Foamsil	Pittsburg-Corning	10		Air	760	ASD-TDR-62-215	
9	X226-32A Foam	American Latex	2	< 400°F	Air	760	G/D Rept. MRG-202	
10	Zirconia A Fiber	H. I. Thompson	8	3000°F	Air	760	Hitco Report AF-33(657)-8902	
11A	ADL-17	A. D. Little	12		Air	760	Bell Survey Data	
11B	ADL-17	A. D. Little	12		Air	0.5	Bell Survey Data	
12A	Fiber Frax Blanket	Carborundum	6.0	2300°F	Air	760	Carbor. Survey Data	
12B	Fiber Frax Paper	Carborundum	9.5	2300°F	Air	760	Carbor. Survey Data	
13	Micro-Quartz Type II & Platinum Foils		6.2 0.67	2750°F	Air	8	G/D A Calc. Data	

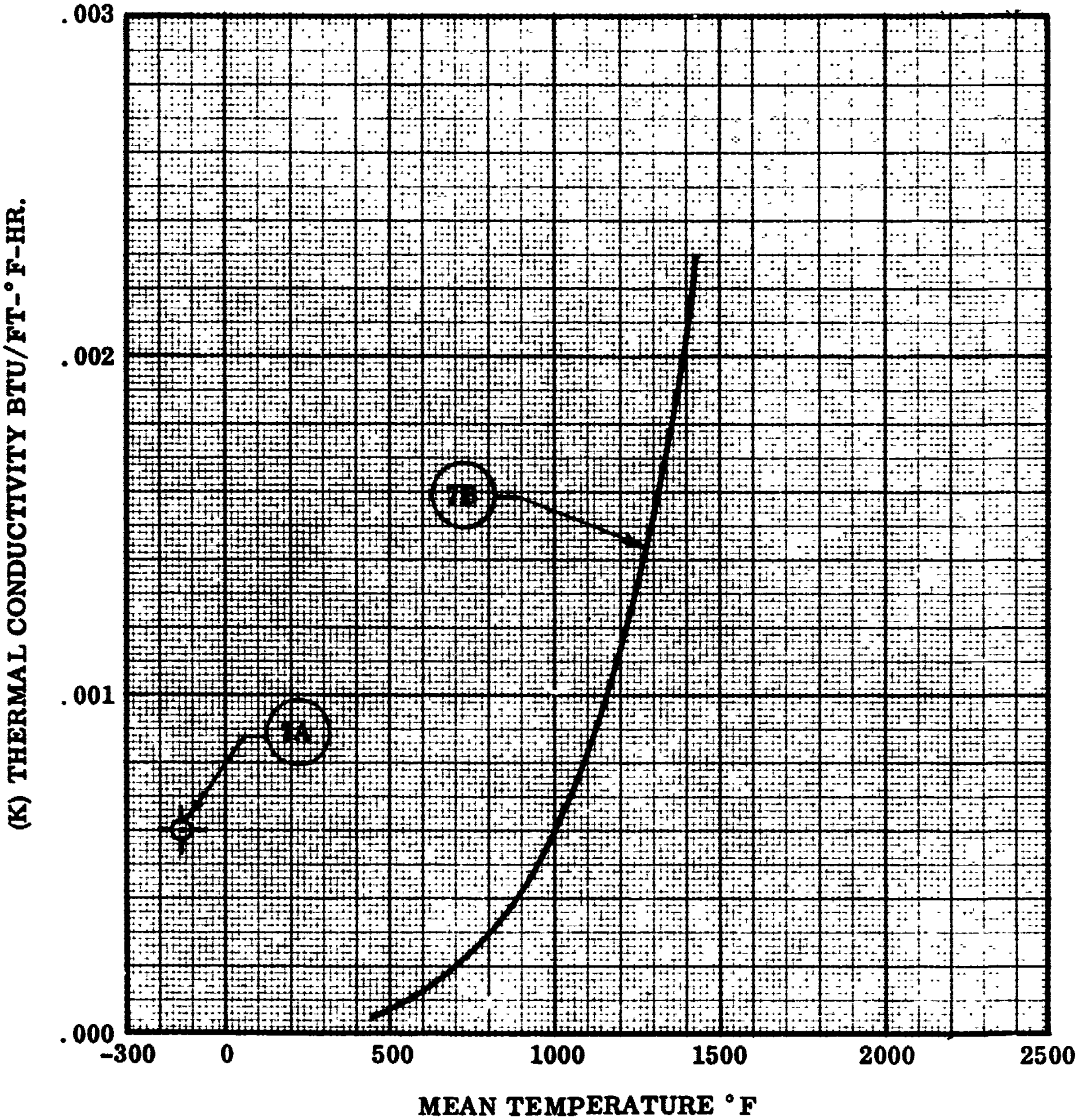

Figure 3. Low Thermal Conductivity Versus Temperature

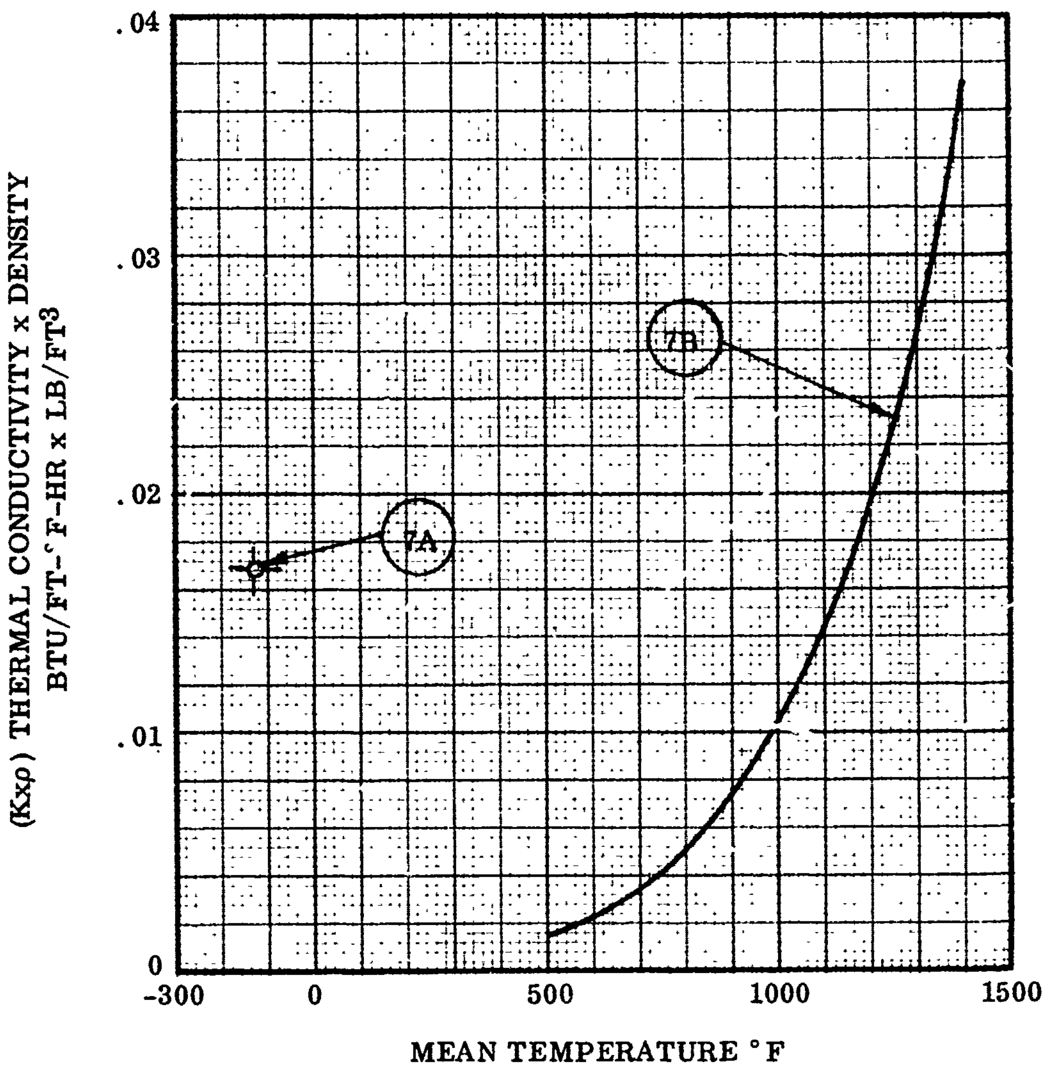

Figure 4. Low Thermal Conductivity Times Density Versus Temperature

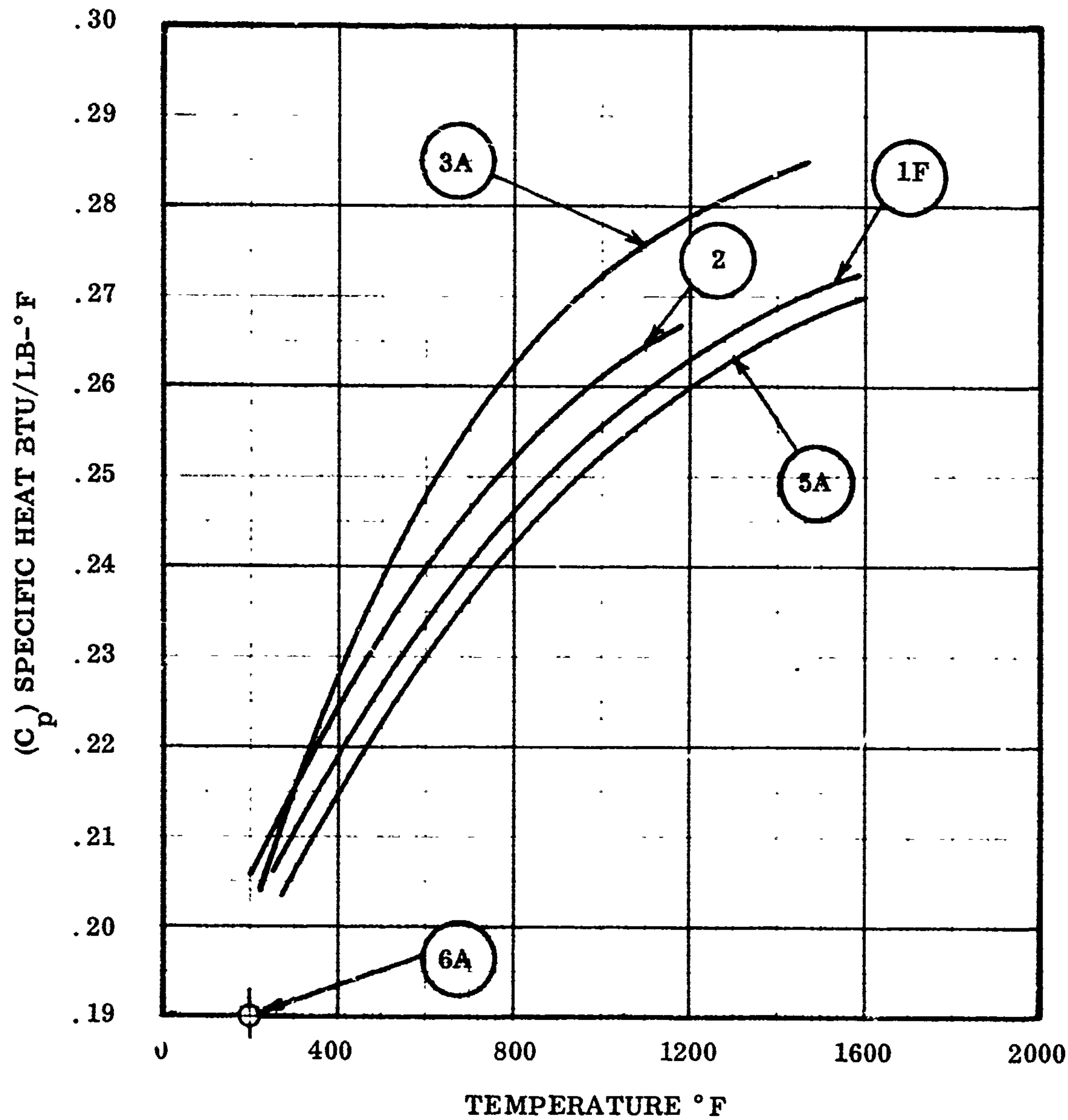

Figure 5. Heat Capacity Versus Temperature

SECTION 3

MECHANICAL ELEMENT MATERIALS

The materials included in this section, as listed in Table 2, have been discussed in Volume I under Composite Designs, and are the materials selected for the Thermal Protection System Test Program under Contract AF 33(657)-9444 during the follow-on phase for 1963.

The selection of Hastelloy C for the cover panel supports was based on its low conductivity and satisfactory strength up to 1800°F. However, the higher oxidation resistance of Hastelloy X, under the same temperature environment, strongly influenced its choice as cover panel material.

Columbium D-36 was selected based on minimum gages limitations and availability for use in temperatures up to 2500°F. Columbium B-66 is being investigated for the program on the basis of its superior creep properties, as compared to Cb D-36 alloy. Both columbium alloys, under the proposed temperature profile, will require protection against oxidation. The results from "Evaluation of Coated Refractory Metal Foil, Contract AF 33(657)-9443," by Solar, a subsidiary of International Harvester Company, have been used to select the appropriate coatings for the columbium alloys.

Chromium composites were selected as material for the columbium cover panel fasteners. Chromium composites have shown excellent oxidation resistance within the temperature range considered for the test program. Its inability to be rerolled in thin foils limited its application to nuts and bolts.

Table 2. Mechanical Element Materials List

PRODUCT NAME	DENSITY LBS/IN.^{3}	SUPPLIER	PROPERTY DATA SOURCE		
			K	α	F_{TU} & F_{TY}
Hastelloy X	.297	Haynes-Stellite	H-S Lit.	H-S Lit.	H-S Lit.
Hastelloy C	.323	Haynes-Stellite	H-S Lit.	H-S Lit.	H-S Lit.
Columbium D-36	.286	Du Pont			Du Pont Lit.
Columbium B-66	.305	Westinghouse		N.A.A. S/D 62-1211	GD/FW Rep.
Chrome Composite	.238	Bendix		Bendix Lit.	Bendix Lit.

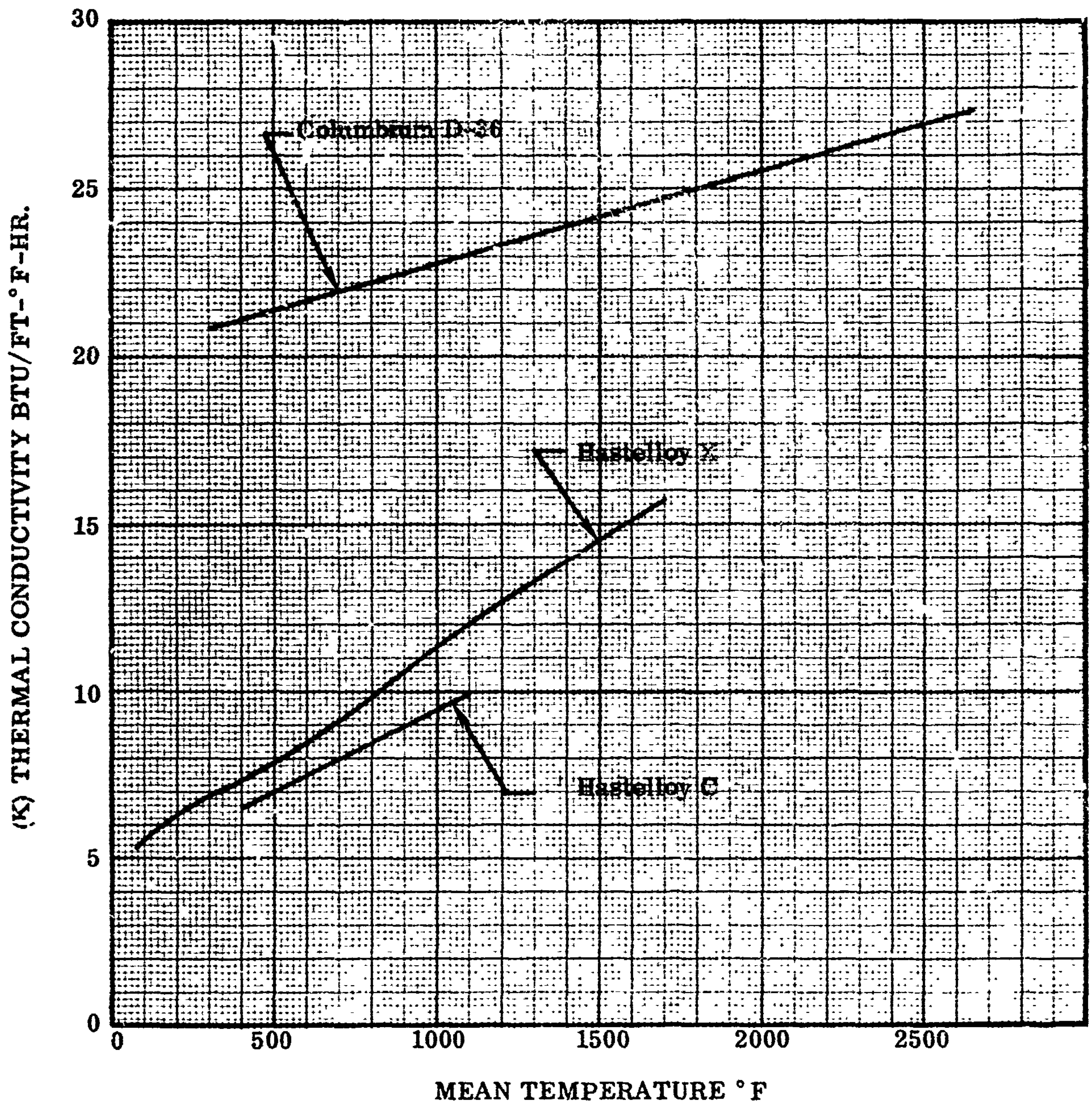

Figure 6. Metal Thermal Conductivity Versus Temperature

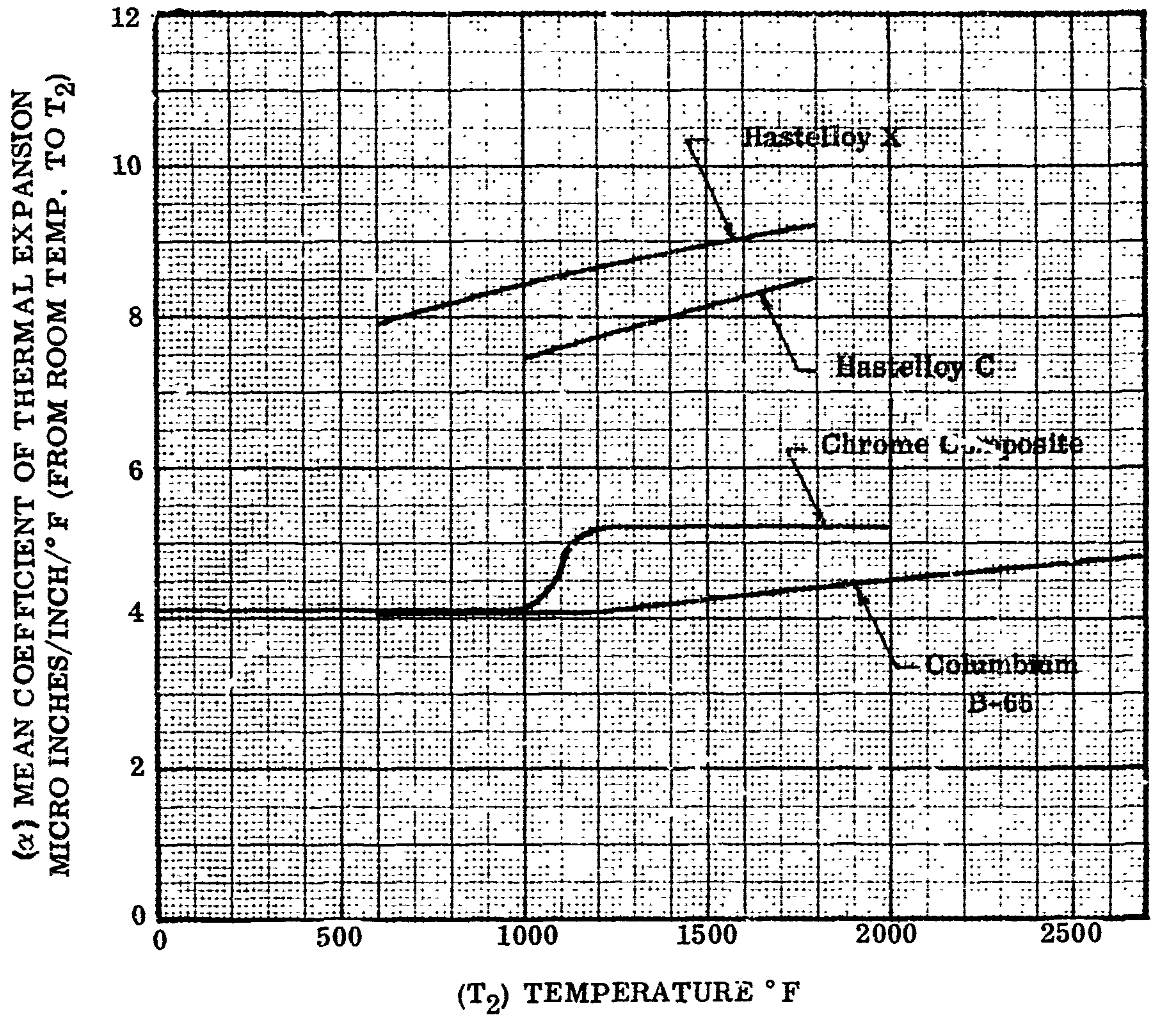

Figure 7. Coefficient of Thermal Expansion Versus Temperature

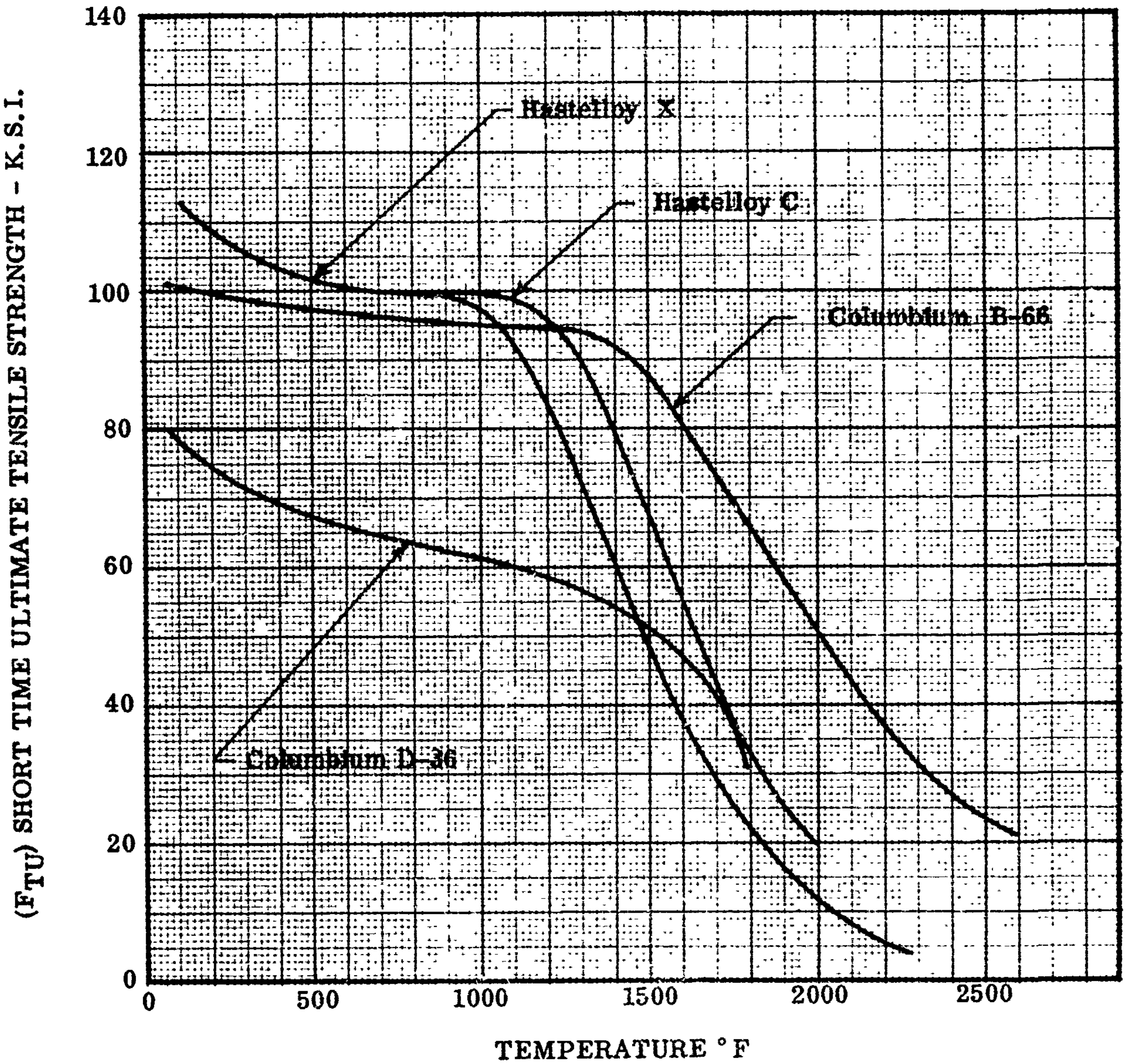

Figure 8. Ultimate Tensile Strength Versus Temperature

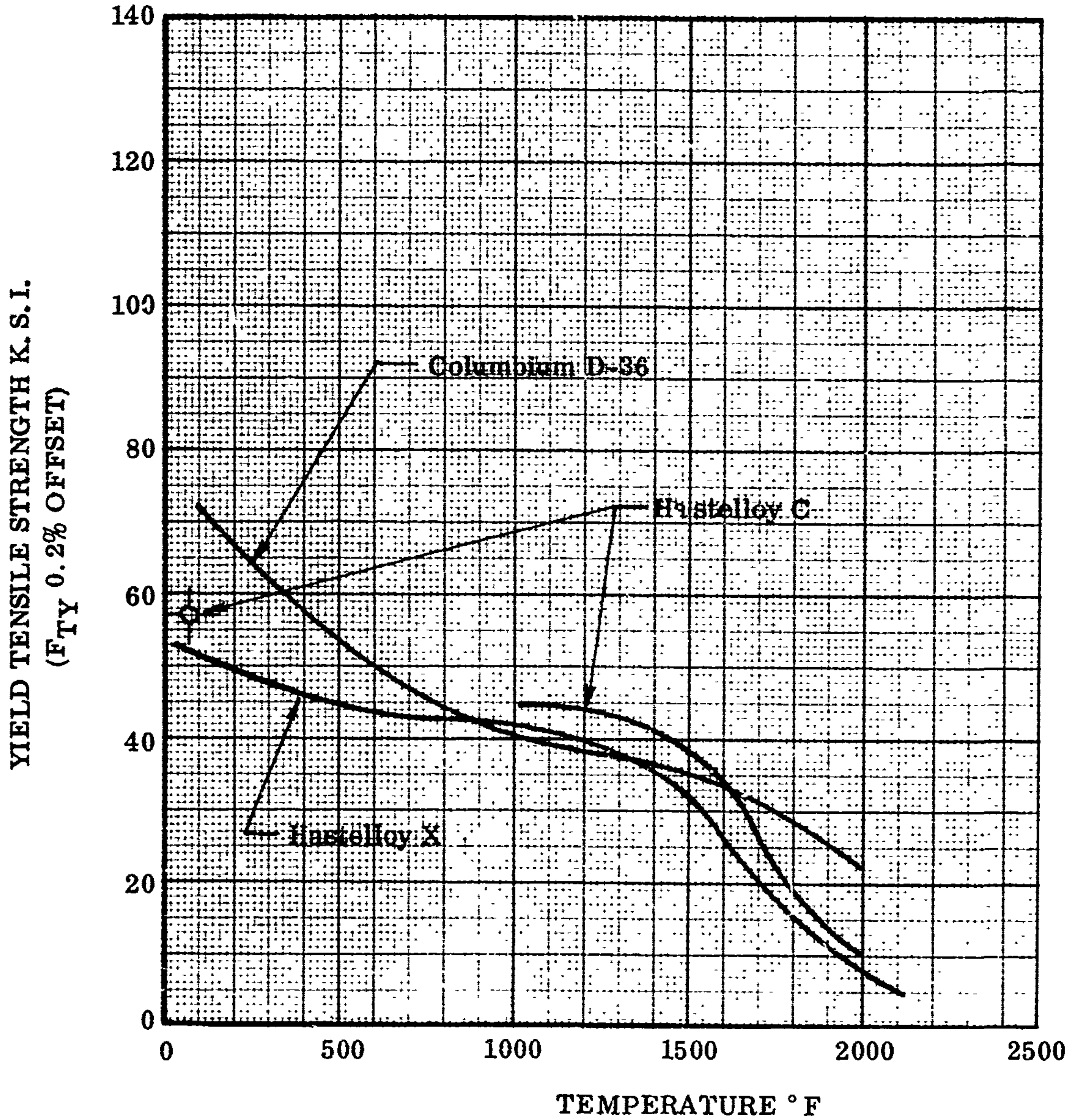

Figure 9. Yield Tensile Strength Versus Temperature

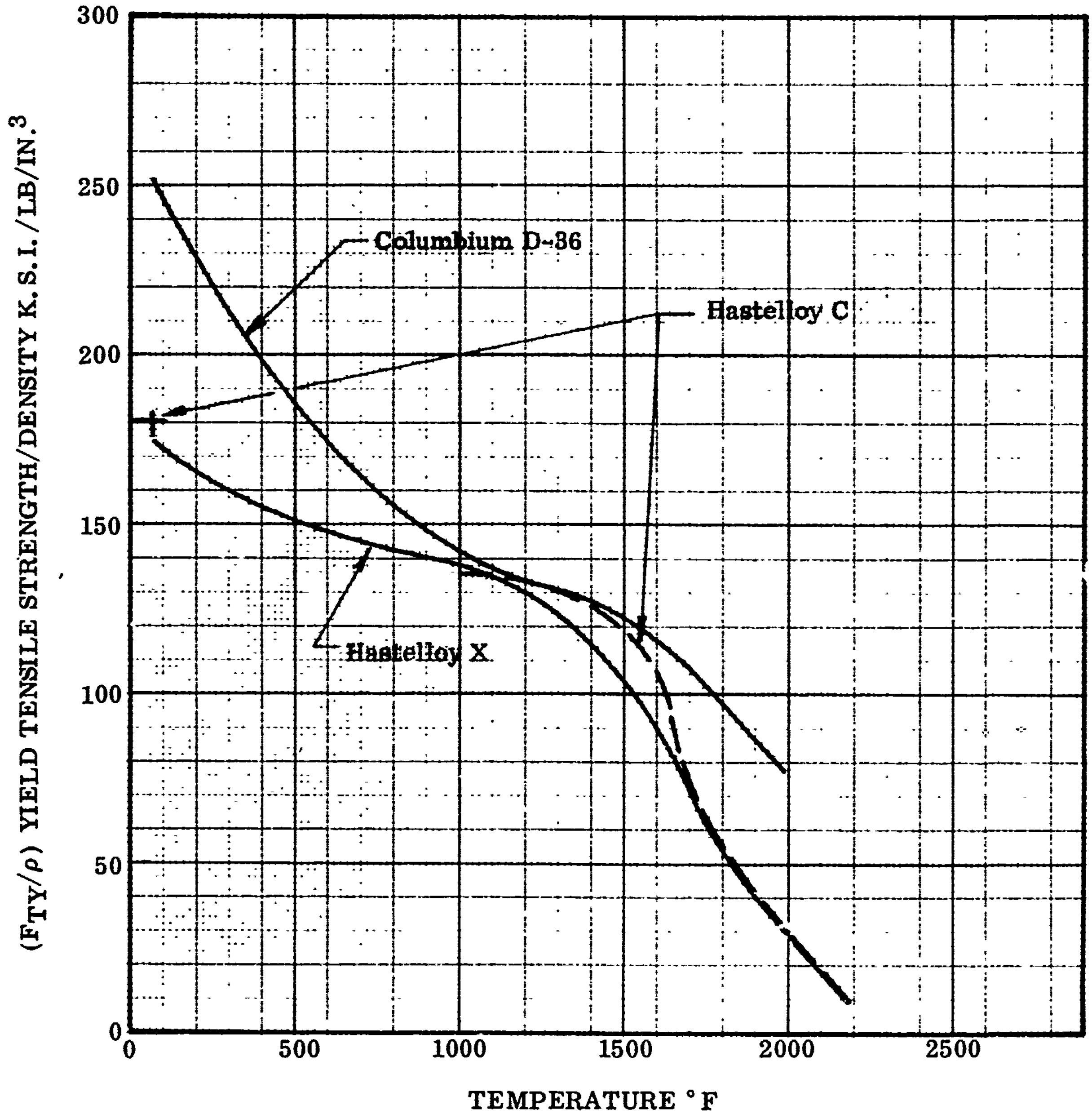

Figure 10. Yield Strength/Density Ratio Versus Temperature

This page intentionally left blank

APPENDIX

THERMAL CONDUCTIVITY OF MICRO-QUARTZ

APPENDIX

THERMAL CONDUCTIVITY OF MICRO-QUARTZ

The data presented in this appendix was supplied to General Dynamics/Astronautics by the Dynatech Corporation of Cambridge, Massachusetts. The majority of the information was extracted verbatim from Dynatech Report No. 408, dated 15 June 1963, which was prepared by J. D. Plunkett.

I. 1 RADIAL THERMAL CONDUCTIVITY APPARATUS FOR PARTICULATE MATERIALS

I. 1. 1 Introduction. Dynatech Corporation has developed an apparatus to measure high temperature thermal conductivities of insulations. The Dynatech TCP-100 measures the thermal conductivities of powders, fibers, and cellular materials in air, inert gas, or vacuum throughout the temperature range -200°F to +3000°F. The design accuracy of this apparatus was ±10%, which we believe has been achieved or exceeded. Furthermore, design features provide significant thermocouple and heater protection as well as incorporating a number of useful and convenient features.

In designing an apparatus to measure the thermal conductivity of particulate materials and insulation systems generally, the single most significant factor in determining the apparatus configuration is the fact that effective thermal insulators possess thermal conductivities as low as can be achieved, lower than can be found for any material per se. Thus, it is impossible to insulate samples in such a way as to produce heat flow only in the desired direction. The sole exception to this statement is the spherical heat flow method which is extremely difficult to use with particulate materials. To illustrate, if a temperature gradient is impressed on the ends of a rod of metal, and the metal is placed in an insulating material, the heat flow down the axis of the rod can be used as a measure of thermal conductivity, since the radial flow can be reduced to an insignificant fraction of the longitudinal flow by the insulators which have thermal conductivity orders of magnitude lower than metals.

For samples such as powdered, cellular, and fibrous materials, the rod method is obviously impractical, simply because it is not possible to obtain materials with lower conductivities, much less one of a conductivity several orders of magnitude less. Even if a material with a nearly infinite thermal resistivity were available, a method would be needed to measure its thermal conductivity. The room temperature thermal conductivity values for various materials provide insight into the problem. Most metals fall into the region of conductivities 0.1 to 1.0 Cal, sec^{-1}, cm^{-2}, $°C^{-1}$, cm; and most solid ceramics fall in the region 0.005 to 0.1 Cal, sec^{-1}, cm^{-2}, $°C^{-1}$, cm. In comparison, the effective thermal conductivity of powders, which are representative of insulating materials, are generally in the region $1 \times 10^{-6} \times$ to 1×10^{-3} Cal,

sec^{-1}, cm^{-2}, $°C^{-1}$, cm., and can be expected to be considerably lower than this for some samples, temperatures, and conditions.

Another property of powder or fibrous insulation is that it is a loose material and thus determines the form of equipment to be used. This means that not only must the sample itself be contained, but also the heaters and thermocouples must be self-supporting. In any event, it is impossible to depend on the sample itself for support. Other considerations that enter into the choice of methods are, volume of the sample, accuracy, and temperature range desired. If samples are expensive or difficult to procure, it is desirable to have a small volume.

I. 1.2 Radial Method. A radial heat flow method was chosen to meet the desired objectives. Heat is generated within an inner heater by electrical resistance and flows out radially through the cylindrical annulus between two concentric cylinders, which contains the sample. This arrangement can be designed in a manner which allows quick and convenient access to the sample, as well as to all heaters and thermocouples.

Providing there are no end losses, the thermal conductivity canbe calculated by the following expression:

$$k = \frac{Ei}{T_1 - T_2} \left[\frac{\ln r_2/r_1}{2 \pi L} \right]$$

Where, k = Thermal conductivity of the sample

E = Voltage drop across the chosen length (L)

i = Electrical current

r_1 = Radius of the inner sample surface

r_2 = Radius of the outer sample surface

T_1 = Temperature of the inner sample surface

T_2 = Temperature of the outer sample surface

All the equipment parameters enclosed in parenthesis constitute a body factor (N). This leaves four experimental quantities E, i, T_2, and T_1, which are required for each data point. The body factor can be set up to express conductivity in any desired units. However, in all experiments at Dynatech Corporation the factor is expressed in the units compatible with Cal, sec^{-1}, cm^{-2}, $°C^{-1}$, cm.

Even though the end losses are significant, it is still possible to determine the conductivity of insulating samples. If a plot of ($\triangle$T) versus (Ei) curve yields a

straight line, then the slope can be used to calculate the sample conductivity, and the intercept of th (Ei) axis gives the magnitude of the non-radial flow.

I. 1. 3 Apparatus Description

I. 1. 3. 1 Test Section. The Dynatech TCP-100 Radial Thermal Conductivity Apparatus is a complete measurement system consisting of a test section, suitable instrumentation and an environmental chamber. The 12-inch long test section is made from two concentric alumina tubes of high purity alumina wound with platinum-rhodium resistance windings.

The inner heater is basically a 1-inch O.D. tube composed of two parts; 1.) an inner core and 2.) a covering sleeve. The 40 mil resistance winding is wound into a spiral groove on the convex surface of the inner core. The inner tube extends down through the sample cell to a mounting flange. The covering sleeve is axially grooved to accept thermocouples on the exterior surface. Heater winding on the inner core contains voltage taps to measure the power dissipated within a 3-inch length of the central zone of the test section.

Bottom and top alumina discs are placed at the ends of the test section. The bottom disc is inserted between a separation in the inner heater sleeve which in turn supports all the sample cell except the inner heater core. An outer 3-inch I. D. auxiliary outer heater surrounds the full 12-inch length of the test section and allows independent control of heat flux and temperature. The outer heater brings the complete test section to approximately the desired temperature, while the inner heater provides the thermal flux to produce a thermal gradient through the sample. Separate power controls are provided for both the inner and outer heaters.

Because the test cell is not sufficiently long to reduce the axial gradients to a sufficiently small value to provide pure radial heat flow, two end guard heaters are employed. These heaters are platinum-rhodium windings embedded in pure alumina slip and wound over alumina tubes. Separate power controls provide independent control for each guard.

Immediately surrounding the outer heater is a 2-1/2-inch thick layer of insulation contained within a water or liquid nitrogen cooled shroud. The shroud is removable to allow access to the test cell. The insulation recommended and usually used is a 100 mesh alumina powder. This insulation is poured in the top and is removed through a suitable port in the shroud base plate. For determination of conductivities near room temperature (-250°F to +500°F) no insulation is used between the shroud and the outer heater. However, the top and bottom guard heaters must be provided with a winding of fibrous insulation to reduce the power requirements of these heaters.

I. 1. 3. 2 Instrumentation. The thermal flux flowing radially through the 3-inch central zone of the test section is determined by measuring the voltage drop along this section and the electrical current through it. Precision AC meters (± 1/4%) are used for both measurements. All power requirements for the apparatus is provided by a stabilized voltage control.

Temperature measurements are made with platinum - 10% rhodium thermocouples from room temperature to 3000° F and copper constantan thermocouples below room temperature. Provision is made for sixteen couples of each type. The outer sleeve of the inner heater contains three grooves approximately 1/16-inch deep and 5/16-inch wide. Into these grooves are placed the couples which are contained inside twin bore 1/16-inch diameter alumina tubing. In a similar manner three couples are mounted on the inner surface of the outer heater. The couple wire is 10 mils in diameter. After a butt bead is produced, the wire is threaded into position through the tube bore. This procedure of mounting the couples within protective tubes at the sample surfaces greatly reduces thermocouple contamination and greatly facilitates handling particularly for powders and fibrous samples. The temperature drop between the thermocouple and the sample surface has been found to be negligible in most cases. For samples which are rigid or semi-rigid, substitute couples can be mounted directly into the sample since a finite sample interface is present and possess a discrete temperature resistivity.

Three thermocouple beads are positioned at the midlength point of both inner and outer heaters. Usually two couple beads on the inner heater are placed one-inch above and below the three-inch central zone. In addition, two couples are positioned one inch in from each end of the sample cell. These couples are used for temperature measurement and control of axial gradients. This leaves two couples for spares or placement within the sample if this proves desirable.

All thermocouples are taken to an ice-water reference junction. Measurements are made with a precision potentiometer, which can be read to 1μ V and is probably accurate to $\pm 3\mu$ V.

Suitable panel ammeters and voltmeters are provided for all four heaters to allow reasonably accurate setting of the manual controls. In addition, controllers can be used to control the temperature of the outer heater and the top and bottom end guard heaters.

I. 1. 3. 3 Environmental System. An environmental system is available which can ultimately attain a range of pressures from 1 atmosphere to 10^{-6} torr with air, inert gas, or other gases which do not affect the noble metal heaters or thermocouples. A mechanical pump, diffusion pump, baffle, chamber and plumbing are available as well as a vacuum gauge and a needle valve controlled leak for pressure control from 10^{-3} torr to 10 torr. Above this pressure the system was pumped to the required pressure and sealed.

I.2 SAMPLE

I.2.1 General. Two specimens, each approximately 12 in. × 14 in. × 1 in. of fibrous silica "Micro Quartz" were received at Dynatech Corporation and were labeled G.D.-1. The density of this material as received was 4.58 lb., ft^{-3}. In order to measure the thermal conductivity normal to the plane of the larger dimensions, eight cylindrical segments 12-inches long were cut from the sheet specimens. This task was accomplished by an insulation specialist and not by Dynatech. Primarily because of the extreme accuracy desired and the delicacy of the operation, special cutting equipment and experience were required.

I.2.2 Body Factor

$$N = \frac{\ln r_2/r_1}{2\pi L}$$

Where: $r_1 = 0.530$ in.

$r_2 = 1.470$ in.

$L = 3.00$ in.

$N = 5.11 \times 10^{-3}$

Note: Dimensions are made to express k as Cal, sec^{-1}, cm^{-2}, $°C^{-1}$, cm.

I.3 TEST PROCEDURE

I.3.1 Sample Mounting. Once the apparatus has been basically assembled, then only sample mounting and instrumentation hook-up is required. This procedure is a relatively short and simple operation. The exact procedure for sample mounting depends upon the nature of the sample. Powders are poured into the sample annulus, while fibers can be compressed down into the annulus if they are loose and unmatted. Fiber mats are mounted by winding the fibers unto the inner heater. When high sample densities of fibers are required, it has been found convenient to wrap the sample with polyethylene sheet secured with celluloid tape. This allows the outer heater to be slid down over the sample with a minimum of sample compression in the bottom region of the test cell.

Rigid or semi-rigid samples are mounted in one of two alternate ways. Either torroids or cylindrical pieces can be cut from sheet materials and mounted. Torroids allow the evaluation of thermal conductivity within the plane of the sheet. Cylindrical segments, usually eight (8), allow measurement of conductivity normal to the plane of the sheet. The cutting and forming of insulations into these shapes is not simple or easy, thus the cylindrical method is a bit restrictive for the measurement of rigid samples.

In the case of rigid or semi-rigid samples it is recommended that thermocouples be mounted directly into the surface or interior of the sample. This procedure is used in all Dynatech measurements as the preferred procedure, though it does require some additional effort.

The G. D.-1 cylindrical segments were assembled around the inner 1-inch heater and the eight pieces fitted together well with absolutely no radial gaps. When the outer heater was slid down over the sample, the whole eight pieces were slightly compressed which maintained the samples in intimate contact. Thermocouples were installed in slots made in the sample to accommodate the 1/16-inch diameter twin base alumina tubing which contained the noble metal wires. The location of all thermocouples was accurately determined to approximately 1/64-inch.

I.3.2 Equipment Operation. After the sample is mounted, most samples are heated to 1,000°F for two hours in air to burn out any organic binders present. Subsequently, the temperature is adjusted to the first measurement which is always the lowest temperature point required. Additional data points are taken with increasing temperature until the highest temperature data is obtained. Subsequent check points can be made on cooling if desirable.

If data is desired as a function of some particular gas pressure, the first temperature is attained at the highest gas pressure with other pressure data points taken with descending pressure. For the second temperature the reverse procedure is followed. Thus, every other temperature data is taken in the same pressure sequence.

After all data is taken the sample is photographed before dismounting and measurements taken to ascertain the shrinkage observed. For vacuum tests, all samples are cooled under vacuum to observe visible sample alteration.

I.3.3 Experimental Procedure. The TCP-100 Apparatus can be operated in one of two modes of operation; as a one-point or as a two-point device. For one point determinations of thermal conductivity at a particular mean sample temperature, the outer heater power is adjusted to bring its temperature to a value approximately 1/2 ΔT less than the average sample temperature sought. The inner heater power is adjusted to give the (ΔT) desired. (This can be determined from a quick calculation using estimated conductivity data). Top and bottom guard heater power is adjusted to yield an isotherm down the inner heater. This operation is rather slow until operator experience is gained.

Two-point, (multipoint) operation is somewhat simpler but yields about the same total experimental information since more data is required. Usually two points are taken; the first at zero inner heater power. That is, the outer heater is adjusted

to provide a desired temperature. Then the inner and outer sample temperatures determined at zero power to the inner heater. With end losses present the inner temperature will be lower than the outer heater, producing a negative radial gradient.

The second point is attained at the same inner heater temperature and the power to the outer heater is adjusted downward while simultaneously sufficient power is applied to the inner heater to produce a zero gradient. In a somewhat similar manner, additional points can be obtained. The slope of the straight line plot of (ΔT) versus inner heater power (Ei) all at the same inner heater temperature yields the value of thermal conductivity. This value of conductivity is an averaged value over several slightly different average sample temperatures. This fact should not cause appreciable error unless the value of conductivity of the sample undergoes very large changes with temperature.

Two, or multipoint data can be taken by keeping the average sample temperature constant and varying the temperature of the inner heater. In general, the absolute value of the end losses increases with temperature up to approximately 1,300°C, then goes through a maximum followed by a minimum about 100°C higher. Since in the high temperature region axial losses are fairly constant above 1200°C, it is recommended that the mean sample temperature be kept constant, and below this temperature the inner heater temperature be held constant. In most cases very little difference in experimental data is observed between these two procedures.

Differences in conductivity values using one-point or two-point procedures are not expected to be appreciable, though we have not investigated these differences as of this time. While in point of fact, both methods are about equally productive of data, we recommend the one-point method of operation. The two-point procedure is useful should a guard heater fail during a test run.

I.4 RESULTS - SAMPLE G.D.-1 (See Figure 11 for graphical presentation.)

I.4.1 1 ATM Air

I.4.1.1 TCP-200 Apparatus

TEMP °C	BTU / FT HR °F	TEMP °F
281.3	3.651×10^{-2}	540
569.8	5.320×10^{-2}	1,057
830.4	7.036×10^{-2}	1,527
857.2	7.327×10^{-2}	1,575
1,162.2	12.380×10^{-2}	2,124
1,394.7	19.682×10^{-2}	2,542

I.4.1.2* Plate Tester Results

TEMP °C	BTU / FT HR °F	TEMP °F
386.7	4.087×10^{-2}	728
392.2	4.062×10^{-2}	738
393.9	4.111×10^{-2}	741

I.4.2 10MM Air

TEMP °C	BTU / FT HR °F	TEMP °F
304.7	2.515×10^{-2}	580
570.0	3.893×10^{-2}	1,058
890.9	5.561×10^{-2}	1,636
871.5	5.392×10^{-2}	1,602
1,200.2	9.648×10^{-2}	2,192
1,403.1	15.645×10^{-2}	2,558

I.4.3 100μ Air

TEMP °C	BTU / FT HR °F	TEMP °F
310.8	1.548×10^{-2}	591
274.4	1.572×10^{-2}	526
579.9	2.660×10^{-2}	1,076
889.5	4.425×10^{-2}	1,634
1,203.8	7.472×10^{-2}	2,198
1,405.6	11.268×10^{-2}	2,562

I.4.4 $< 10^{-3}$ Air Residue in Vacuum

TEMP °C	BTU / FT HR °F	TEMP °F
316.7	1.420×10^{-2}	602
584.3	2.321×10^{-2}	1,084
892.7	3.941×10^{-2}	1,639
1,209.5	6.964×10^{-2}	2,209
1,408.1	10.905×10^{-2}	2,567

*Check points run on a Dynatech Model TC-2000 Guarded Hot Plate Apparatus.

I.4.5 1 ATM Helium

TEMP °C	BTU / FT HR °F	TEMP °F
294.3	5.150×10^{-2}	562
566.9	9.043×10^{-2}	1,052
903.7	12.719×10^{-2}	1,659
1,171.6	22.173×10^{-2}	2,141
1,363.2	41.469×10^{-2}	2,486

I.4.6 10MM Helium

TEMP °C	BTU / FT HR °F	TEMP °F
296.8	3.651×10^{-2}	566
572.5	3.368×10^{-2}	1,062
870.3	7.061×10^{-2}	1,599
871.6	7.665×10^{-2}	1,601
1,182.9	12.477×10^{-2}	2,161
1,405.8	18.087×10^{-2}	2,562

I.4.7 100μ Helium

TEMP °C	BTU / FT HR °F	TEMP °F
307.5	2.007×10^{-2}	585
595.9	3.361×10^{-2}	924
888.3	5.417×10^{-2}	1,631
1,217.5	8.947×10^{-2}	2,223
1,406.6	13.202×10^{-2}	2,564

I.5 OBSERVATIONS AND CONCLUSIONS

I.5.1 General Sample Alternation During Test. The sample did not undergo any visible color change while in the testing apparatus. The general appearance of the sample did not indicate significant melting or alteration. The eight sample segments were lightly sintered together. As could best be determined from general handling, finger nail scratching, rubbing, and bending, the sample was in "excellent" condition with remarkably little changes from the original material. Of the many samples we have tested, this sample maintained its original general appearance and properties better than any other material.

I.5.2 Sample Shrinkage. During the course of the test program, the highest temperature maintained for a point was approximately 2700°F on the inside of the test sample cylinder. However in attaining equilibrium at the highest temperature, the

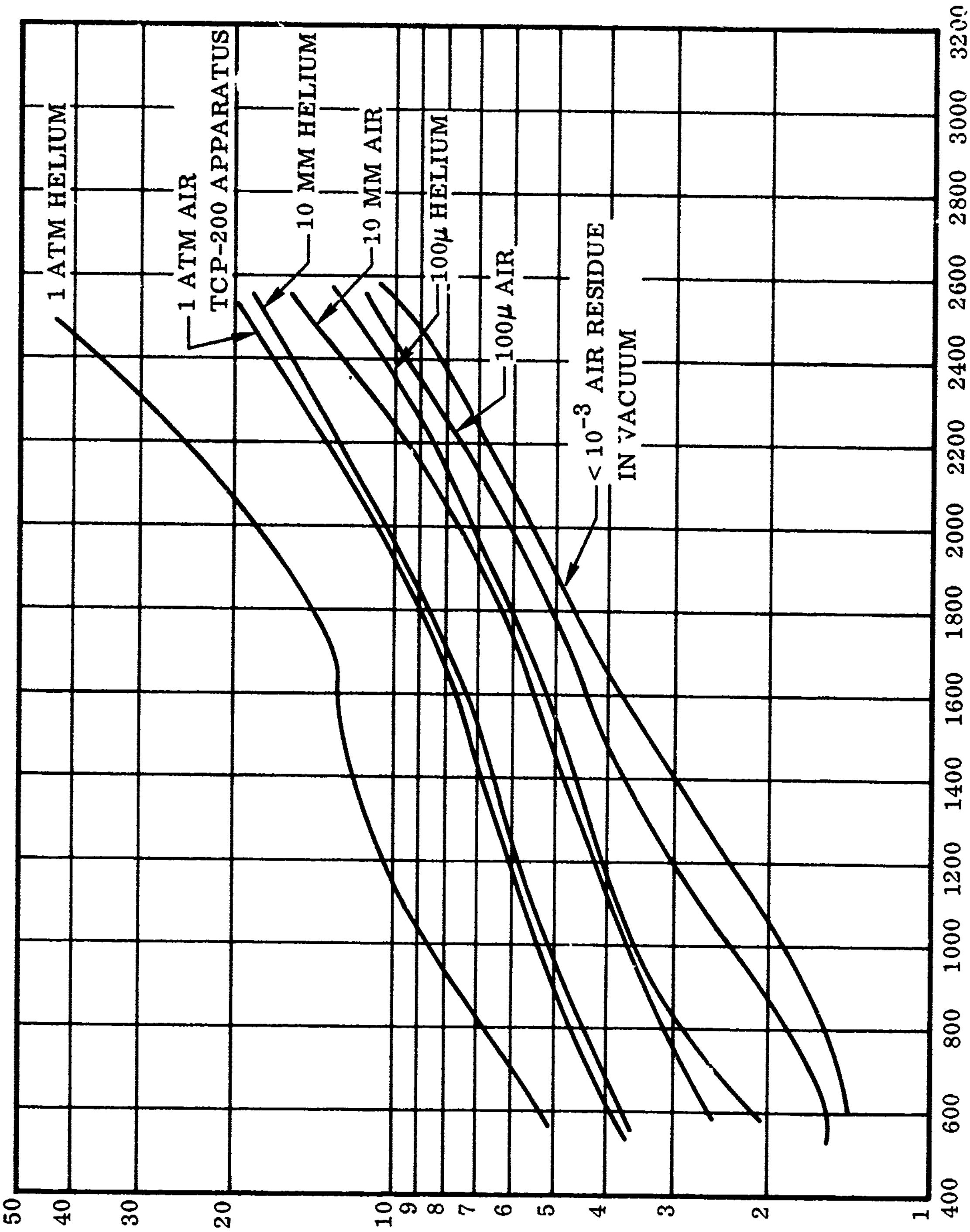

Figure 11. Thermal Conductivity Versus Temperature, Results-Sample G.D.-1

inside heater was incorrectly adjusted and its temperature rose to approximately 2900°F overnight. This appears to have been the only cause of shrinkage in the sample since neither the external diameter nor the length were altered while the internal diameter increased by 1/8 inch. This is equivalent to a shrinkage of approximately 2.5%.

Thus we believe that up to the maximum sample equilibrium temperature attained, 2700°F, no significant sample shrinkage was observed. Again we should like to point out that this sample performance as regards the slight shrinkage was the best, i.e. the least change in dimensions, we have observed.

I.5.3 Conclusions. As regards low thermal conductivity, dimensional stability, freedom from visible deterioration, this sample was superior to all samples we have tested. We should like to observe, however, that most of our prior experience has been with flexible fiber mats rather than rigid insulations of a type similar to G.D.-1.

Aeronautical Systems Division, AF Materials Laboratory, Wright-Patterson AFB, Ohio.

Rpt Nr ASD-TDR-63-596, Vol. II. LIGHTWEIGHT THERMAL PROTECTION SYSTEM DEVELOPMENT: Materials Existing Data and Recommended Data Acquisition (U). Interim Report, June 63, 34 pp. incl illus, tables.

Unclassified Report

This is a summary technical report covering the first two phases of a program leading to the development of a lightweight thermal protection system. This volume describes the known properties of insulation materials and makes recommendations for acquiring additional data. Volume I of this report contains design criteria, a description of composites, and an outline of the test program.

1. Thermal insulation
2. Insulating materials
3. Data

I. AFSC Project 651G
II. Contract AF 33(657)-9444
III. General Dynamics/Astronautics, San Diego, Calif.
IV. R.A. Lange, et al
V. Secondary Rpt Nr GDA63-0123-2
VI. In DDC collection

Aeronautical Systems Division, AF Materials Laboratory, Wright-Patterson AFB, Ohio.

Rpt Nr ASD-TDR-63-596, Vol. II. LIGHTWEIGHT THERMAL PROTECTION SYSTEM DEVELOPMENT: Materials Existing Data and Recommended Data Acquisition (U). Interim Report, June 63, 34 pp. incl illus, tables.

Unclassified Report

This is a summary technical report covering the first two phases of a program leading to the development of a lightweight thermal protection system. This volume describes the known properties of insulation materials and makes recommendations for acquiring additional data. Volume I of this report contains design criteria, a description of composites, and an outline of the test program.

1. Thermal insulation
2. Insulating materials
3. Data

I. AFSC Project 651G
II. Contract AF 33(657)-9444
III. General Dynamics/Astronautics, San Diego, Calif.
IV. R.A. Lange, et al
V. Secondary Rpt Nr GDA63-0123-2
VI. In DDC collection